Effect of Deposition Parameters on MCrAl Coating Properties Using Dual Magnetron Sputter Deposition

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Abstract

Dual magnetron sputter deposition was used to prepare MCrAl coatings with various compositions (Fe-Ni-Cr-Al or Ni-Cr-Al). One magnetron was used for MCr while the other for pure Al. The MCr targets were made of commercial grade Ni-20Cr, 304 stainless steel (SS) and 310SS. Two deposition technologies were employed including conventional magnetron sputter deposition and plasma enhanced magnetron sputter deposition (PEMS) deposition, in which a global plasma was generated from hot filament thermionic electron emission. The effect of deposition parameters on the coating morphology, microstructure, composition and adhesion were studied extensively using a number of techniques including SEM, EDS, XRD and Rc indentation. It has been observed that the sputter deposited coatings are typically nanocrystalline with the average grain size being about a few tens of nanometers. Under selected deposition conditions, the coatings are very dense with nearly no appearance of columnar structure. They have excellent adhesion to the substrates. Dense, continuous and stable Al$_2$O$_3$ oxide layers have been observed after oxidation at 750°C and 1010°C in separate studies. The oxide layers also show excellent spallation resistance. In this paper, the deposition processes are discussed and the coating characterization results are presented.

Introduction

Components of steam and gas turbines and boilers for power generation operate at high temperatures in oxidizing environments. MCrAl or MCrAlY (M = Fe, Ni, Co or the combination) coatings are commonly used for the protection of the components in industry [1-4]. Typically, these coatings are prepared using various spray processes [5-8], but physical vapor deposition (PVD) techniques, including electron beam evaporation, triode sputtering, arc plating and magnetron sputtering, have attracted significant attention particularly in recent years [9-19]. This is because the grain sizes of the coatings produced using PVD methods are much finer than their counterpart spray coatings, typically from sub-micrometers to a few tens of nanometers. Fine grain structured, especially the nanostructured, coatings are more resistant to high-temperature oxidation and corrosion. Among the PVD techniques, magnetron sputter deposition may be the most commonly used method [10-16, 20-24], because it is fairly easy to prepare nanocrystalline coatings with multi-elements such as Fe-Ni-Cr-Al-Y under various compositions.

Two methods for magnetron sputtering were reported previously on the preparation of micro-/nano-structured coatings. In one common method, the target was made from vacuum melting and casting from pure metals with the desired composition of MCrAlY (M = Fe, Ni, Co or the combination, while Y may or may not be needed). In the other method, Al slugs were embedded into a metallic target that was made from AISI 310SS to obtain the Fe-Cr-Ni-Al coating [14]. In both methods, only one target was used for the deposition. While both methods are simple in preparing a coating with a specific composition, it is difficult to vary the composition, especially the Al concentration, once the target has been made. If the Al concentration is to be varied to understand its effect on oxidation and for optimization, many targets have to be prepared.

The PEMS process is a variation of magnetron sputtering. The major difference is the introduction of a global plasma, independent of the magnetron plasma, which is generated in the entire vacuum chamber by electron thermionic emission from tungsten filaments. Before the coating deposition, the ions from the global plasma are drawn to the surface of the samples via a bias voltage to sputter clean the surface from residual contaminants and native oxide. At this time, the magnetrons are not turned on, unlike in the conventional magnetron sputtering process in which magnetrons have to be turned on to generate plasma for the sample cleaning, and as a result deposition of the target materials may occur before the samples are cleaned. After the cleaning of the parts in the PEMS process, the magnetrons are turned on for the coating deposition while the global plasma is maintained. The ion flux delivered to the sample surface is greatly enhanced from the global plasma. Consequently, both the adhesion and the density of the coating are increased. Detailed discussion of the PEMS process can be found elsewhere [25-29]. The PEMS process has been developed for the deposition of super hard, nitride-based nanocomposite coatings [25-29]. The objective of this paper is to determine whether or not PEMS can be used to deposit MCrAl coating. In this paper, we will discuss the experimental results obtained from sputtering of various MCr targets with Al. A study of the microstructure, composition and adhesion of the coatings will be presented, while the oxidation behavior of the coatings will be presented in separate publications [30, 31].

Experimental Procedures

Material Selection and Coating Deposition Using the PEMS Process

In this study, two planar magnetrons were used, one for Al and the other for MCr (M = Fe, Ni, or the combination). For MCr targets 304SS, 310SS and Ni-20Cr were used to process various compositions of Fe-Ni-Cr-Al and Ni-Cr-Al coatings. All the MCr target materials were commercial grade, while the Al target had a purity of 99.995%. The nominal composition of each MCr target material is listed in Table 1. The targets were 170 mm (6.75”) in diameter by 9.5 mm (3/8”) thick. The specimens to be coated were made of three materials including AISI 304SS, Haynes 230 and P91. In this paper, we only report the study on 304SS, while the results from Haynes 230 and P91 can be found in Refs 30 and 31. The specimens were machined to rectangular blocks of 17.8 mm x 12.7 mm x 3.1 mm (0.70” x 0.50” x 0.12”), and then ground and polished to 6 μm finish using standard metallographic techniques. At each end of the block a hole was drilled so multi-specimens were able to be chained and deposited at the same time. They were cleaned in an ultrasonic bath in acetone and then alcohol before installed in the vacuum chamber for processing. Figure 1 shows a photograph of the experimental setup, in which multiple samples are being deposited with a 310SS+Al coating. The left magnetron is Al target with a low magnetron power, while the right one is a 310SS target with a high magnetron power. Two tungsten filaments are used for generating the global plasma, in addition to the plasmas generated in front of the magnetrons. The samples were chained on a double rotation fixture to obtain uniform deposition.
Table 1. Chemical composition of targets selected for deposition of the MCrAl coatings.

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Composition (wt.%)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
</tr>
<tr>
<td>310SS (Target for coating)</td>
<td>Bal.</td>
</tr>
<tr>
<td>Ni20Cr (Target for coating)</td>
<td>-</td>
</tr>
<tr>
<td>304SS (Target for coating &amp; specimen)</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Before the deposition, the global plasma was generated at 10 A of discharge current for ion cleaning of the specimens with the parts being biased at 120 V. The cleaning time was 120 minutes. The long duration of cleaning was used mainly to prevent the samples from re-oxidation due to the out-gassing of water moisture inside the vacuum chamber. After the cleaning, without interruption, the deposition started by turning on the magnetrons while both the bias voltage and the filament power were adjusted to the specified value to achieve the ion assisted deposition. The MCr magnetron power was maintained at 4 kW for most tests, while the Al power was varied from 0 to 1.3 kW, to obtain various concentration of Al. In this paper we only present the data obtained from Al=0.55 kW and 1.1 kW. The deposition parameters are shown in Table 2. The three coatings that were studied are 310SS+Al, Ni20Cr+Al, and 304SS+Al. Listed in Column 1 is the test number, and Column 2 the sample ID. Listed from Column 3 through Column 6 are the target material and the corresponded magnetron power. Column 7 lists the discharge current $I_d$. When the global plasma is not used as in the conventional magnetron deposition, it is 0 A. Columns 8 and 9 show the sample bias voltage $V_b$ and the bias current $I_b$, which correlates to the ion flux. Column 10 lists the deposition time.

As can be seen from Table 2, the tests are divided into four groups. In the first group (310SS+Al coating), Test Nos. 1-3, no filament generated global plasma is used ($I_d$=0A), while three bias voltages ($V_b$=0, 100 and 150 V) are studied. In the second group (also 310SS+Al coating), Test Nos. 4-6, two magnetron power levels ($P_1$ and $P_2$), two discharge currents bias voltages ($I_d$) and two bias voltages ($V_b$) are studied. In the third and fourth groups, Ni20Cr+Al, and 304SS+Al are studied. In both cases, two discharge currents ($I_d$=0A and 15A) are used to study the effect of ion bombardment. The deposition time for all depositions is varied according to the magnetron power to achieve the targeted coating...
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thickness of 30–40 μm. However, the deposition of Test No. 3 (DE8) was terminated early due to an unexpected facility interruption, but the samples were still good for analyses.

Specimen Characterization

After the deposition scanning electron microscopy (SEM) was used to examine the surface morphology and cross-sectional structures. Energy dispersive X-ray spectroscopy (EDS) was used for the composition characterization. X-ray diffraction (XRD) was carried out to determine the coating phases and the grain size. The adhesion of the coatings were evaluated using a qualitative standard Rc indentation test at 150 kg load that is widely accepted by the research community and industry [32-34]. The crack formation around the crater is examined. The adhesion of a coating can be ranked into six categories. For ranks “1” and “2”, cracks can be observed in the coating without coating delamination. For ranks “3” and “4”, the coating may be spalled near the indent at only a couple of locations. For rank “5”, more spallation occurs. But for rank “6”, the coating surrounding the crater is all delaminated. A coating with the ranking from “1” through “4” is deemed acceptable.

Results and Discussions

Deposition Rate, Composition and Grain Size

The experimental data are listed in Table 2 from Column 11 through 14. The coating thickness, obtained from the cross-sectional SEM micrographs, is about 30-40 μm, which corresponds to a deposition rate of ~7-10 μm/h when the filament-generated global plasma was not turned on ($\text{I}_\text{g}=0\text{A}$). The only difference among these three tests was the bias voltage. As can be seen, when $V_b=0\text{V}$ (Sample ID No. DE7), i.e. no ions were drawn to the specimen during the deposition, the coating shows a very loose columnar structure. Many defects form near the interface. As the film grows thicker, the columns grow wider. When $V_b=100\text{V}$ (DE1), a small flux of ion can be drawn from the magnetron-generated plasma ($\text{I}_\text{g}=0.11\text{A}$). At this time the coating shows a “cauliflower”-like morphology, and the cross-section shows it is a dense coating without the typical columnar structure. When the bias voltage is further increased to 150V (DE8), a slightly high current is obtained ($\text{I}_\text{g}=0.15\text{A}$), but a similar structure to that for $V_b=0\text{V}$ is obtained, except that the feature columnar grain size is slightly smaller. It appears that too high ion energy has destroyed the dense structure. Based on these three tests, the 310SS+Al coating deposited at $V_b=100\text{V}$ has the best structure.

Morphology and Microstructure of 310SS+Al Coatings

Conventional magnetron sputter deposition

A number of tests were conducted using 310SS and Al targets to form 310SS+Al coatings. Shown in Figure 2 are the topological (left) and cross-sectional (right) views of the 310SS+Al coatings deposited using conventional magnetron sputter deposition (Test Nos. 1-3 in Table 2), i.e. the filament-generated global plasma was not turned on ($\text{I}_\text{g}=0\text{A}$). The only difference among these three tests was the bias voltage. As can be seen, when $V_b=0\text{V}$ (Sample ID No. DE7), i.e. no ions were drawn to the specimens during the deposition, the coating shows a very loose columnar structure. Many defects form near the interface. As the film grows thicker, the columns grow wider. When $V_b=100\text{V}$ (DE1), a small flux of ion can be drawn from the magnetron-generated plasma ($\text{I}_\text{g}=0.11\text{A}$). At this time the coating shows a “cauliflower”-like morphology, and the cross-section shows it is a dense coating without the typical columnar structure. When the bias voltage is further increased to 150V (DE8), a slightly high current is obtained ($\text{I}_\text{g}=0.15\text{A}$), but a similar structure to that for $V_b=0\text{V}$ is obtained, except that the feature columnar grain size is slightly smaller. It appears that too high ion energy has destroyed the dense structure. Based on these three tests, the 310SS+Al coating deposited at $V_b=100\text{V}$ has the best structure.

Plasma enhanced magnetron sputter deposition

Shown in Figure 3 are the topological (left) and cross-sectional (right) views of the 310SS+Al coatings deposited using the plasma enhanced magnetron sputter deposition process (Tests Nos. 4-6 in Table 2), i.e. the filament-generated global plasma was used during the film growth. From Table 2, $\text{I}_\text{g}=10-15\text{A}$, $V_b=40-60\text{V}$ and $\text{I}_\text{g}=0.8-1.25\text{A}$. At this time the ion flux during the film growth is 7-10 times that used in Test Nos. 1-3.

When deposited at $\text{I}_\text{g}=10\text{A}$ and $V_b=40\text{V}$ (DE2, Figure 3a), the coating shows a loose columnar structure (cross-sectional view). Although the coating seems to have a smaller feature size and denser structure (top view) than that shown in Figure 2b ($\text{I}_\text{g}=0\text{A}$, $V_b=100\text{V}$), it is still full of defects. Figure 3b shows the SEM photographs of DE4, which was deposited using the same discharge current and bias voltage as for DE2.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Sample ID</th>
<th>Target 1 Material</th>
<th>Target 2 Material</th>
<th>Pt (kW)</th>
<th>Target 2 Material</th>
<th>P2 (kW)</th>
<th>Id (A)</th>
<th>Vb (V)</th>
<th>Ib (A)</th>
<th>Deposition Time (h)</th>
<th>Coating Thickness (μm)</th>
<th>Deposition Rate (μm/h)</th>
<th>Al in Coating (μw%)</th>
<th>Grain Size (nm)</th>
<th>Microstructure</th>
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</tr>
<tr>
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<td>0</td>
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<td>4</td>
<td>33.1</td>
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<td>7.7</td>
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<td>100</td>
<td>0.11</td>
<td>4</td>
<td>33.8</td>
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<td>Al</td>
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<td>310SS</td>
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<td>150</td>
<td>0.15</td>
<td>2</td>
<td>13.1</td>
<td>6.6</td>
<td>12.2</td>
<td>9.8</td>
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<td>0.80</td>
<td>4</td>
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<td>8.8</td>
<td>13.6</td>
<td>15.7</td>
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</tr>
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<td>5</td>
<td>DE-4</td>
<td>Al</td>
<td>0.55</td>
<td>310SS</td>
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<td>1.28</td>
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<td>38.5</td>
<td>7.7</td>
<td>11.6</td>
<td>21.7</td>
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<tr>
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<td>Ni20Cr</td>
<td>4</td>
<td>0</td>
<td>100</td>
<td>0.14</td>
<td>4</td>
<td>35.1</td>
<td>8.8</td>
<td>11.0</td>
<td>9.5</td>
<td>Columnar</td>
<td></td>
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<tr>
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<td>DE-10</td>
<td>Al</td>
<td>1.1</td>
<td>Ni20Cr</td>
<td>4</td>
<td>15</td>
<td>60</td>
<td>1.38</td>
<td>4</td>
<td>27.8</td>
<td>7.0</td>
<td>8.9</td>
<td>24.1</td>
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<td>Group 4: 304SS+Al, Coating</td>
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<td>10</td>
<td>DE-12</td>
<td>Al</td>
<td>1.1</td>
<td>304SS</td>
<td>5</td>
<td>0</td>
<td>100</td>
<td>0.10</td>
<td>4</td>
<td>41.0</td>
<td>10.3</td>
<td>11.7</td>
<td>12.3</td>
<td>Dense</td>
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<tr>
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<td>DE-13</td>
<td>Al</td>
<td>1.1</td>
<td>304SS</td>
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<td>15</td>
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<td>1.14</td>
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<td>7.3</td>
<td>10.8</td>
<td>36.3</td>
<td>Dense</td>
<td></td>
</tr>
</tbody>
</table>
but the magnetron power levels for both Al and 310SS were all reduced to one half, while the deposition time was increased to reach about the same coating thickness. The feature size is reduced while the columns become smaller. Figure 3c shows the SEM photographs of DE3, which was deposited at $I_d=15\, \text{A}$ and $V_b=60\, \text{V}$, the heaviest ion bombardment. At this time, the surface "cauliflower" feature size becomes even smaller while the cross-section view shows the coating is very dense.

Based on the 310SS+Al study and comparing the microstructure of samples DE1. (Figure 2b) and DE3 (Figure 3c), it can be concluded that these two sets of deposition parameters would produce dense and nearly columnar free structures. They are the conventional magnetron sputter deposition at 100V bias and the PEMS process at 60V bias with 15A discharge current.

**Morphology and Microstructure of Ni20Cr+Al Deposited Samples**

Based on the 310SS+Al coating data, two deposition trials were conducted using the Ni20Cr and Al targets to form the Ni20Cr+Al coatings. The deposition conditions for Ni20Cr+Al coatings (Tests Nos. 7 and 8) are listed in Table 2. Similar to the 310SS+Al study, we also used the conventional magnetron sputter deposition at 100Vb (DE9) and PEMS continued on page 34.
Effect of Deposition Parameters
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process at 60Vb with 15A discharge current (DE10). The SEM images are shown in Figure 4. Comparison of the microstructure of the two samples shows that the deposition using the PEMS process at $V_b=60V$ and $I_d=15A$ resulted in a dense coating without columnar structure.

Morphology and Microstructure of 304SS+Al Deposited Samples
Two deposition trials were conducted using the 304SS and Al targets to form the Fe18Cr8Ni+Al coatings. The deposition conditions for 304SS+Al (Test Nos. 10 and 11) are listed in Table 2. Similar to the deposition of 310SS+Al and Ni20Cr+Al trials, both conventional magnetron sputter deposition at 100Vb (DE12) and PEMS process $V_b=60V$ and $I_d=15A$ (DE13) were studied. The SEM images are shown in Figure 5. Comparison of the cross-sectional images reveals that the microstructures of both samples are similar, but the coating surface morphology of DE13 is slightly better than DE12. For DE13, the grain boundaries of the coating are not as clear as those for DE12.

Adhesion Testing
Shown in Figure 6 are the representatives of Rc indents for selected 310SS+Al and Ni20Cr+Al coatings deposited under two sets of condi-

![SEM images of 304SS+Al coatings](image1)

![SEM images of 310SS+Al coatings](image2)

Figure 3. Effect of ion bombardment on morphology (left) and cross-sectional (right) microstructure of 310SS+Al coatings using the PEMS process.
Figure 4: Morphological (left) and cross-sectional (Right) SEM images of Ni20Cr+Al coatings.

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tions: without or with the global plasma. Using the standard, all coatings fall within rank “1” and rank “2”, all showing good adhesion. Careful examination of the indents and considering the microstructure results presented above reveal that the coatings prepared using the PEMS method at \( V_b = 60 \text{V} \) and \( I_d = 15 \text{A} \) show fewer, but slight bigger cracks, while the coatings prepared using conventional magnetron sputtering show more numbers but fainter cracks. This is because the PEMS coatings are denser and are usually harder than the conventional magnetron sputtered coatings \([28, 29]\). In contrast, coatings with columnar structures and defects have fewer cracks after the Rc indentation because the softer coatings with more defects can absorb more plastic energy.

XRD Study

X-ray diffraction spectroscopy was used to study the phase formation of the coatings for selected samples. The diffraction patterns of 310SS+Al coatings are shown in Figure 7. The XRD spectra for only two samples representing most coatings are shown. Figure 7a shows the coating deposited using conventional magnetron sputtering (\( V_b = 100 \text{V} \) and \( I_d = 0 \text{A} \)), while Figure 7a shows the coating using the PEMS process (\( V_b = 60 \text{V} \) and \( I_d = 15 \text{A} \)). Beside the major peaks Fe-Cr observed from both samples, other peaks are observed including Ni-Cr-Fe, AlNi, and AlFe from the sample DE1. It seems that the high ion bombardment using the PEMS process suppresses the formation of some of these phases as compared with the conventional magnetron sputtered coating (DE1). Using the width of the most intense XRD peak, the average grain size can be estimated and the data are listed in Table 2. As can be seen from the results presented in the table, the grain size of the deposited MCrAl coatings are about 10-25 nm. To more accurately identify the phase formation and estimate the grain size and evaluate the microstructure, orientation image microscopy and transmission electron microscopy (TEM) work are being conducted on selected samples.

Based on the microstructure, adhesion and XRD data from all three coatings (310SS+Al, Ni20Cr+Al and 304SS+Al), the conventional magnetron sputter deposition at \( V_b = 100 \text{ V} \) and the PEMS process at \( V_b = 60 \text{V} \) and \( I_d = 15 \text{A} \) were selected for the deposition of the nano coatings on samples for oxidation and corrosion testing \([30, 31]\).

Conclusion

Dual magnetron sputter deposition has been used to prepare MCrAl or MCrAlY coatings with various compositions (FeNiCrAl and NiCrAl). These sputter deposited coatings are typically nanocrystalline with the average grain size being of a few tens of nanometers. The deposition parameters have a significant effect on the coating morphology and microstructure. Under selected deposition conditions, the coatings are very dense with nearly no appearance of columnar structure. They have excellent adhesion to the substrates. Dense, continuous and stable oxide layers have been observed after oxidation tests up to 1010 °C \([30,31]\). The oxide layers also show excellent spallation resistance. Although further research and development are still needed, it is believed that these coatings are suitable for application on steam and gas turbine components that operate at elevated temperatures.
Figure 6. Rc indentation of MCrAl coatings. (a) 310SS+Al, $I_d = 0A, V_b = 100V$; (b) $I_d = 15A, V_b = 60V$; (c) Ni20Cr+Al, $I_d = 0A, V_b = 100V$; and (d) Ni20Cr+Al, $I_d = 15A, V_b = 60V$.

Figure 7: X-ray diffraction spectra of MCrAl coatings for (a) 310SS+Al, $I_d = 0A, V_b = 100V$; (b) $I_d = 15A, V_b = 60V$.

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35. Ronghua Wei is an Institute Scientist, Materials Engineering Department, Mechanical & Materials Engineering Division, at Southwest Research Institute (SwRI), San Antonio, TX. Dr. Wei has been conducting research in the areas of materials science and surface engineering, plasma science and engineering, and tribology since 1981. Since joining SwRI in 2001, Dr. Wei has led a research group in conducting research on plasma surface engineering and developing new technologies for the government and industries. His research is focused on plasma enhanced magnetron sputtering (PEMS) and plasma immersion ion implantation, deposition (PIII&D) and high intensity plasma ion nitriding (HIPIN). His research resulted in the development of a new PEMS technology that won the R&D100 Award in 2009.

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